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# Determination of Volatile Organic Compounds in Fish Samples by Vacuum Distillation and Fused Silica Capillary Gas Chromatography/Mass Spectrometry

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# Determination of Volatile Organic Compounds in Fish Samples by Vacuum Distillation and Fused Silica Capillary Gas Chromatography/Mass Spectrometry

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**A vacuum distillation apparatus and a procedure developed for the analysis of fish tissue samples are described. The volatile organic compounds are distilled from the sample and characterized by gas chromatography/mass spectrometry (GC/MS) using a fused-silica capillary column (FSCC). Examples of volatile organic compounds that can be detected in fish tissue distillates are provided.**

A previous study demonstrated that volatile organic priority pollutants could be quantitatively isolated and concentrated in a cold trap (−196 °C) after vacuum distillation from fish tissue samples (*1*). In that study the distillates were diluted to 5 mL and analyzed as a water sample using Method 624 (*2*). The packed column used in the study was recommended for volatile organic analyses of water (*2*) and had been used in procedures for the analyses of nonwater media (*3*, *4*). Fish tissue samples contain purgeable compounds which adsorb to the packed columns and the desorption of those compounds during column conditioning can take several hours before the next analysis can be started. The extended time between analyses is a costly problem when using packed columns for analysis of a large number of samples.

Fused silica capillary columns (FSCC) provide an attractive alternative approach to packed columns for chromatography of volatile compounds. Since the FSCC can be heated to a higher temperature (350 °C) than that recommended for the packed column, the column preparation time between fish analyses can be greatly decreased. Also, the poor chromatography exhibited by late eluting compounds on packed columns is drastically improved when using FSCC chromatography.

The purge and trap technique is presently used to concentrate volatile organic compounds from a water solution for gas chromatography. Desorption of the volatile concentrate from the trap, however, requires large carrier gas flow rates. The carrier gas flow rate for FSCC is limited to approximately 2 mL/min and, therefore, it was necessary to devise a different technique to concentrate volatile compounds. The vacuum distillation procedure provides a concentrate readily transferred through the FSCC with a minimum flow rate of carrier gas.

This paper demonstrates the use of a vacuum distillation/FSCC procedure for qualitative analysis of fish tissue for volatile compounds. The types of organic compounds that can be detected are illustrated by data obtained in the analysis of fish with this procedure.

## EXPERIMENTAL SECTION

**Chemicals.** Blank water was prepared by first evaporating 100 mL from 1 L of freshly distilled water by gently boiling the water. The remaining boiled water was transferred to a 1-L volumetric flask that had been modified by replacing the ground joint fitting with a 15 mm i.d. O-ring connector. The flask containing the boiled distilled water was then connected to the distillation apparatus at the sample chamber site (see Figure 1). The boiled water was then degassed by evacuating and agitating the flask in an ultrasonic cleaner for 15 min. After evacuation the flask was sealed with a cap made from an O-ring connector, a Buna-N O-ring, and a pinch clamp.

Bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane (Supelco, Inc., Bellefonte, PA), benzene-$d_6$ and

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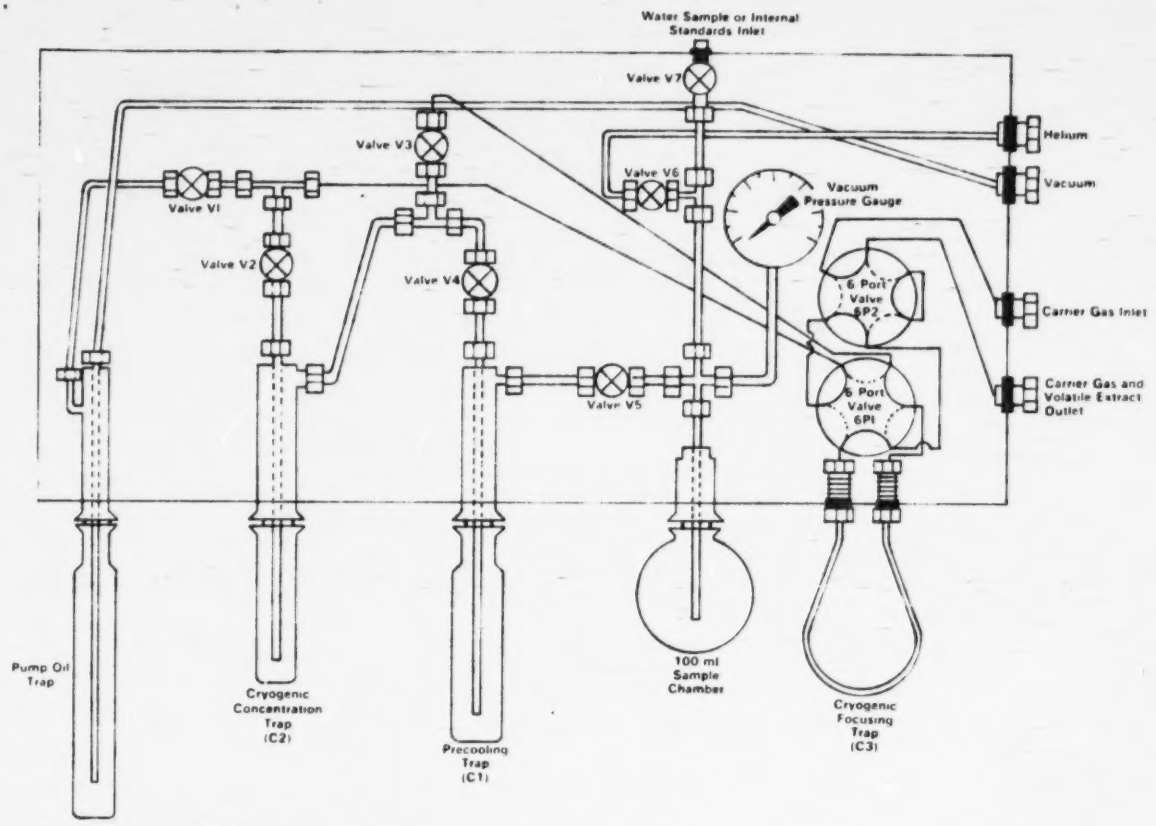


**Figure 1.** Vacuum distillation apparatus.

toluene-$d_8$ (Merck), Inc., Rahway, NJ) were used as internal standards. Standard solutions were prepared in methanol to provide a single stock solution of 2.0 mg/mL of each standard. An internal standard spike solution was prepared by adding 8 μL of the internal standard stock solution to 1 mL of blank water. Internal standard spikes were prepared by adding 5 μL of the internal standard stock solution (80 ng) to 2 mL of blank water contained in a 5 mL gastight luer tip syringe. The sample was spiked with the contents of the syringe immediately before distillation.

The volatile priority pollutant standards were obtained from Supelco, Inc. (Bellefonte, PA). These standards were combined and diluted in ethylene glycol to make one 25 ng/μL standard stock solution. Standard spiking solutions were prepared by adding the desired amount of standard stock solution to 2 mL of blank water containing the internal standard spike.

**Apparatus (See Figure 1).** Volatile organic compounds are desorbed under vacuum from samples placed in the sample chamber. Water contained in the sample chamber is condensed in cold trap C1. Vapors not condensed in trap C1 are condensed in cryogenic trap C2 which is cooled in liquid nitrogen. After distillation is complete, trap C2 is slowly warmed to room temperature and the vapors are recondensed in cryogenic focusing trap C3. Trap C3 is heated to 150 °C and the vapors are flushed with helium carrier gas to the GC/MS. The six-port valves (6P1 and 6P2) direct the transfer of the volatile vapors.

The low pressure used for this study during distillation was supplied by a vacuum pump capable of $10^{-3}$ torr and a 25 L/min flow rate. A glass trap for vacuum pump oil vapors was connected to the $^3/_8$ in. i.d. copper vacuum manifold with 15 mm i.d. O-ring connectors. The trap, cooled in a liquid-nitrogen bath prevented pump oil vapors from back-flushing to trap C2 and prevented water vapors from contaminating the vacuum pump oil.

The transfer lines that connect to the six-port stainless steel valves (6P1, 6P2) were $^1/_{16}$ in. o.d. stainless steel tubing and were connected with $^1/_{16}$ in. o.d. compression fittings. The other transfer lines were glass-lined stainless steel $^1/_4$ in o.d. tubing and connections were made with compression fittings and graphite ferrules. Gastight Nupro B-4BKT toggle values (values V1–V7) were connected to the glass-lined tubing with $^1/_4$ in. o.d. compression fittings and graphite ferrules.

The sample chamber consisted of a 100-mL Pyrex bulb joined to a 15 mm i.d. Pyrex O-ring connector. Trap C1 was a Pyrex cylinder (3.5 cm diameter and 19 cm long) joined to a 15 mm i.d. Pyrex O-ring connector. Modified fittings were used to connect the glass traps (C1 and C2) and the sample chamber to the vacuum distillation apparatus. The modified stainless steel fittings were made by silver soldering stainless steel $^1/_4$ in o.d. compression fitting bodies to 15 mm i.d. stainless steel O-ring connectors. The top soldered compression fitting was drilled to allow the $^1/_4$ in. glass-lined transfer line to extend through the compression fitting/O-ring connector and into the glass traps. Trap C3 and 15 cm of $^1/_8$ in. o.d. quartz tubing bent in a U-shape (Willson Scientific Glass, Arcadia, CA). Trap C3 was connected to the system with $^1/_8$ in. o.d. compression fittings and graphite ferrules. Trap C3 was wrapped with 30 AWG nichrome wire and was heated to 180 °C by electrical current through the nichrome wire controlled with a variable transformer.

A stainless steel box 72 cm long × 10 cm deep × 26 cm high enclosed the valves and transfer lines. Heating tape was wrapped around the transfer lines and valves and was used to maintain the temperature of the vacuum distillation apparatus at 80 °C. The transfer line between the vacuum distillation apparatus and the FSCC was kept at 120 °C. A helium line was connected to the vacuum distillation apparatus at valve V6 to vary pressure in the system and to check for possible leaks. A mechanical pressure gauge was used to measure pressure (0–1000 mmHg).

Extracts prepared by vacuum distillation were analyzed by Finnigan 1020 GC/MS. A DB5 capillary column (30 m long, 0.32 mm i.d., 1.0 μm film thickness) obtained from J & W Scientific, Inc. (Rancho Cordova, CA), was used for chromatography. The gas chromatograph (GC) oven temperature was programmed at −99 °C for 3 min, then increased to 70 °C at a rate of 30 °C/min, and then increased to 180 °C at a rate of 20 °C/min. The GC

temperature program was begun when the transfer of the volatile distillate to the GC was started. Helium carrier gas with a flow of 2 mL/min assisted transfer. The GC effluent was analyzed by the mass spectrometer which was set to scan from $m/e$ 46 to $m/e$ 260 at 1-s intervals.

**Procedure.** The fish samples were prepared from fish delivered to the laboratory under dry ice. Whole fish were chopped at the laboratory with a food cutter (Hobart, 8145 Model, without attachment hub). Liquid nitrogen was added to the samples during chopping to prevent evaporation of volatile organic compounds. Ten grams of homogenized fish tissue was stored in the sample chamber glassware, previously described, and sealed with a Buna-N O-ring, a 15 mm i.d. O-ring connector cap, and a pinch clamp. The sealed samples were stored at −30 °C in a freezer until analyzed.

Residual moisture was removed from the vacuum distillation apparatus by opening valves V1, V2, V4, and V5 and evacuating the sample chamber, trap C1, trap C2, and the joining transfer lines. The evacuation was continued for 7 min after the disappearance of any visible moisture in the glass traps.

Traps C1 and C2 were cooled after residual moisture had been removed from the apparatus. Trap C1 was cooled in a −20 ± 1 °C ethanol bath contained in a Dewar cylinder flask (12 cm wide, 20 cm deep). Trap C2 was cooled with liquid nitrogen (−196 °C) in a Dewar cylinder flask (7 cm wide, 15 cm deep).

After closing valves V2 and V5, a sample storage vessel (sample chamber) containing 10 g of frozen fish sample was connected to a vacuum distillation apparatus and valve V5 was opened. The spike solutions, previously described, were added by attaching the 5-mL syringe to the luer fitting on valve V7. Valve V7 was opened and the sample spike solution pulled into the sample chamber by the vacuum in trap C1, trap C2, and sample chamber. Valve V7 was closed. Valves V1, V4, and V5 were opened and valves V2, V3, and V7 remained closed. Valve V6 was opened to allow helium (5 psi) to enter the sample chamber and adjoining traps C1 and C2 until a pressure of 360 torr was obtained. The sample chamber was immersed in a bath contained in a Bransonic 12 ultrasonic cleaner and agitated for 5 min. An earlier examination demonstrated that continuous agitation of the water bath contained in the ultrasonic cleaner warmed the water to approximately 50 °C.

Transfer of volatile compounds was begun by opening valve V2, and the ultrasonic agitation was continued. The pressure in the sample chamber and trap C1 during vacuum distillation was primarily due to water vapor pressure (~93 torr Hg at 50 °C, 0.78 torr Hg at −20 °C) (5). Vacuum distillation was stopped after 15 min by closing valves V2, V4, and V5.

The volatile compounds concentrated in trap C2 by vacuum distillation were transferred to trap C3 for transfer to the FSCC. Trap C3 was prepared prior to the transfer by turning off the transformer used to heat trap C3 and cooling trap C3 in liquid nitrogen. The six-port valves were in the transfer position (continuous lines in six-port valves as illustrated in Figure 1) and valve V3 was opened. The transfer of the distillate was begun by removing liquid nitrogen bath from trap C2 and allowing trap C2 to warm to ambient temperature. The transfer was assisted by vacuum supplied through the six-port valves (6P1, 6P2) while the distillate was recondensed in cryogenic trap C3.

The transfer of the volatile compounds to C3 was stopped after 15 min by closing valve V3. The six-port valves 6P1 and 6P2 were switched to the injection position (broken lines in six-port valves as illustrated in Figure 1). The GC oven temperature program, as previously described, was started and trap C3 flash heated to 180 °C. The transfer of the concentrate to the capillary column was stopped after 3 min by returning valves 6P1 and 6P2 to the transfer position.

During the distillate transfer (trap C2 to trap C3) the sample chamber and trap C1 were replaced with clean dry units. The sample spike inlet was flushed with helium (5 psi) by opening and then closing valves V6 and V7.

During the GC injection of the distillate, trap C1, trap C2, and the sample chamber were evacuated by opening valves V2, V4, and V5 and closing valve V3. After transfer of the distillate to the fused-silica capillary column was completed and the six-port valves (6P1 and 6P2) were returned to the transfer position, trap C3, heated to 180 °C, was evacuated for 5 min to remove any remaining volatile compounds. The next sample was then introduced and the next distillation begun.

**Table I. Identification and Tailing Factors of Peaks in Figure 3**

| no. | compound | retention time[a] (min:s) | tailing factor[b] |
|---|---|---|---|
| 1 | chloromethane | 7:54 | 1.5 |
| 2 | vinyl chloride | 8:16 | 1.5 |
| 3 | bromomethane | 8:48 | 1.9 |
| 4 | chloroethane | 8:59 | 2.9 |
| 5 | trichlorofluoromethane | 9:26 | 1.6 |
| 6 | acrolein | 9:31 | 3.8 |
| 7 | acetone | 9:36 | 2.1 |
| 8 | 1,1-dichloroethane | 9:49 | 1.1 |
| 9 | acrylonitrile | 9:55 | 5.9 |
| 10 | dichloromethane | 9:58 | 2.3 |
| 11 | *trans*-1,2-dichloroethene | 10:17 | 1.3 |
| 12 | 1,1-dichloroethane | 10:26 | 1.6 |
| 13 | bromochloromethane (internal standard) | 10:49 | 2.0 |
| | chloroform | 10:49 | 2.4 |
| 14 | 1,1,1-trichloroethane | 11:08 | 1.0 |
| | 1,2-dichloroethane | 11:10 | 1.5 |
| 15 | benzene-$d_6$ (internal standard) | 11:17 | 1.5 |
| | benzene | 11:19 | 1.2 |
| | carbon tetrachloride | 11:19 | 1.1 |
| 16 | trichloroethene | 11:43 | 1.4 |
| | 1,2-dichloropropane | 11:43 | 1.3 |
| 17 | bromodichloromethane | 11:50 | 1.6 |
| 18 | 2-chloroethyl vinyl ether | 12:00 | 1.6 |
| 19 | *cis*-1,3-dichloropropene | 12:10 | 1.4 |
| 20 | *trans*-1,3-dichloropropene | 12:27 | 1.1 |
| | toluene-$d_8$ (internal standard) | 12:28 | 1.3 |
| 21 | toluene | 12:30 | 1.4 |
| 22 | 1,1,2-trichloroethane | 12:33 | 1.3 |
| | 2-bromo-1-chloropropane (internal standard) | 12:36 | 1.5 |
| 23 | dibromochloromethane | 12:53 | 1.9 |
| 24 | tetrachloroethene | 12:58 | 1.2 |
| 25 | chlorobenzene | 13:25 | 1.1 |
| 26 | ethylbenzene | 13:35 | 1.1 |
| 27 | *p,m*-xylene | 13:41 | 1.2 |
| 28 | bromoform | 14:01 | 1.6 |
| 29 | *o*-xylene | 14:06 | 1.1 |
| 30 | 1,4-dichlorobutane (internal standard) | 14:21 | 1.2 |
| 31 | 1,1,2,2-tetrachloroethane | 14:27 | 1.0 |

[a] 30 m DB5 fused silica capillary column (0.32 mm bore, 1.0 μm film thickness) preceded by 18 in. of 1/16 in. o.d. stainless steel tubing. Carrier gas helium at 2 mL/min. Temperature program: 3 min isothermal at −99 °C, then 30 °C/min to 70 °C, then 20 °C/min to 180 °C. Hold at 180 °C for 10 min. [b] The tailing factors were calculated as per ref 2 and illustrated in Figure 2.

The volatile distillates were analyzed by GC/MS. Chromatographic peaks were manually selected from the resulting reconstructed ion chromatogram and the compound indentifications were assisted by a computerized forward-search of the EPA/NIH mass spectral library. Many isomers of the same chemical formula could not be distinguished by their mass spectra; therefore the compound with the best matching library spectrum was listed in Tables III and IV.

## RESULTS AND DISCUSSION

The use of vacuum distillation combined with FSCC chromatography has greatly improved the chromatography of the volatile organic compounds contained in fish samples. Improved recoveries of the volatile organic priority pollutant compounds had been demonstrated with vacuum distillation (*1*) and FSCC gave better resolution than packed columns for the late-eluting compounds.

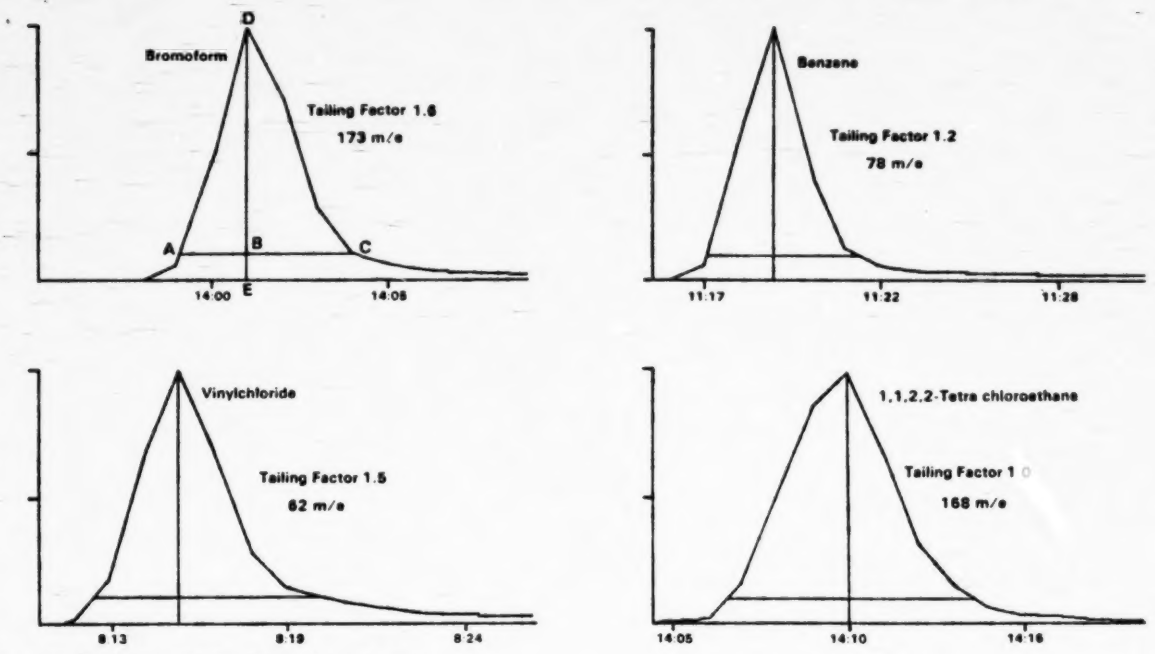


**Figure 2.** Illustration of tailing factor determinations.

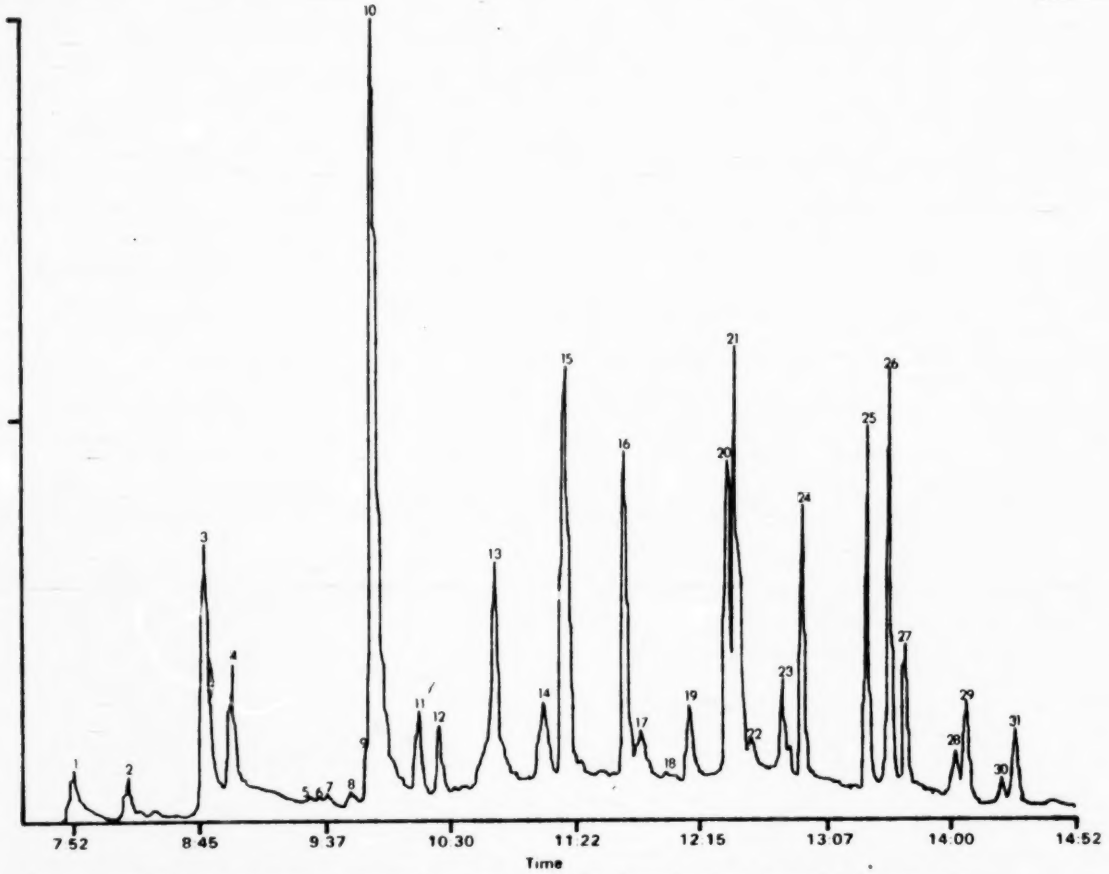


**Figure 3.** Chromatogram of a vacuum distillate prepared from 100 ng amounts of volatile priority pollutants in blank water.

Quality of chromatography was evaluated using the tailing factor described in ref 4 and illustrated in Figure 2. The tailing factor is the result of dividing BC by AB when ABC is a horizontal line drawn at 10% peak height and DBE is a vertical line through the apex (see Figure 2). Tailing factors are a parameter for describing peak symmetry. Symetrical peaks with a tailing factor of 1 are ideal for identification and quantitation. The tailing factors for the volatile organic

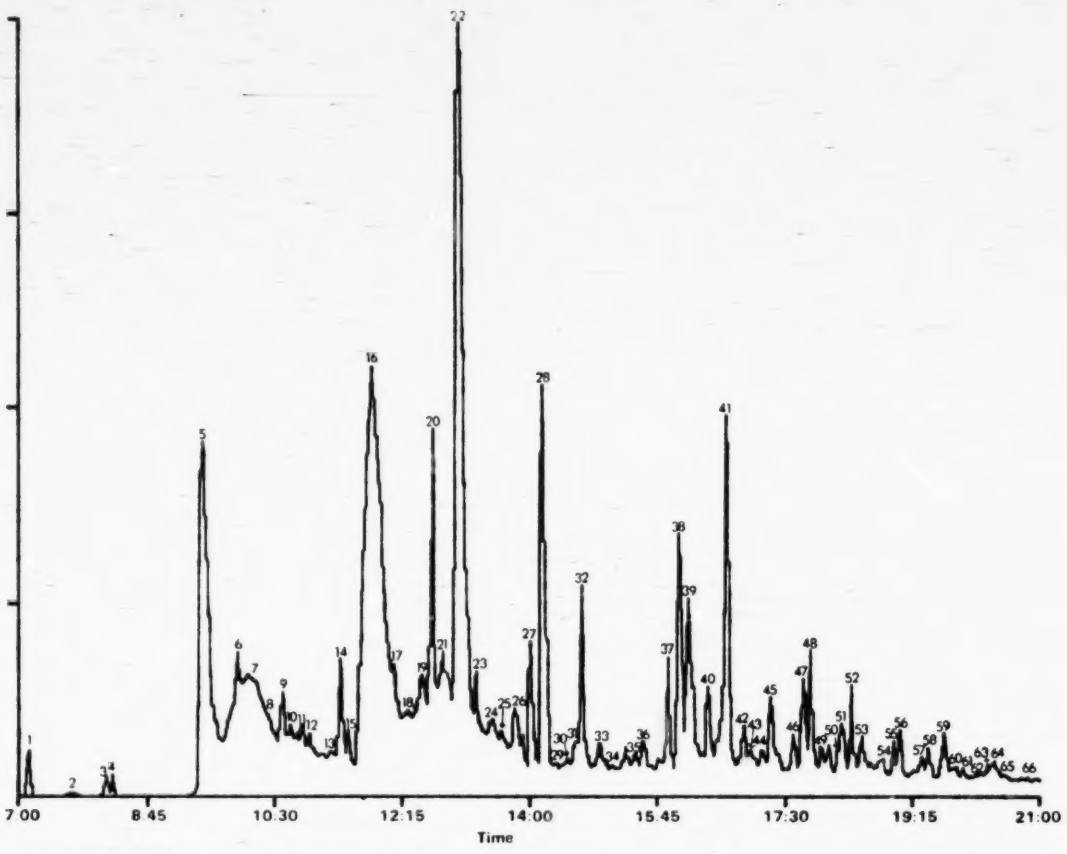


Figure 4. Chromatogram of a vacuum distillate prepared from a 10-g rainbow trout sample.

priority pollutants were determined from the data obtained by analyzing a solution containing 100 ng of each of the volatile organic priority pollutants by this distillation procedure (Figure 3 and Table I). Tailing factor requirements are not indicated in the *Federal Register* for volatile organic priority pollutants; however, a comparison can be made with the tailing factor requirement for semivolatile chromatography (*4*) with 100 ng of benzidine (tailing factor to be less than 3) and 40 ng of pentachlorophenol (tailing factor to be less than 5). For the volatile organic priority pollutants 77% of the tailing factors are less than 1.7. Tailing factors exceeded 3 only for acrolein and acrylonitrile. The tailing factors for late-eluting aromatic pollutants in this sample were less than 2 (naphthalene, 1.2; 4-ethyl-1,2-dimethylbenzene, 0.9; 1,3,5-trimethylbenezene, 1.1; indan, 1.0; propylbenzene, 1.2; and 4-carene, 1.3). Under the same GC conditions direct injection of the priority pollutants resulted in very poor chromatography due to solvent effects. There was little chromatographic improvement when methanol, water, or ethylene glycol was used as a solvent.

The theoretical plates of the volatile priority pollutant compounds were typically between 100 000 and 600 000. Alkyl-substituted aromatic homologues extracted from fish tissue were found to give $N_{sep}$ less than 17.

The chromatograms from the GC/MS analyses of fish distillates are illustrated by Figures 4–8 and the components, which were identified with the assistance of a computerized forward search of the EPA/NIH mass spectral library, are listed in Tables II–IV. The results obtained with this technique for the analysis of a 10-g tissue sample of rainbow trout (taken from the Colorado River below Hoover Dam) are shown in Figure 4 and Table II. The GC/MS results of an abnormally complex distillate obtained from the vacuum distillation of 10 g of fish (carp taken from Las Vegas Wash, NV) tissue are shown in Figure 5 and Table III.

Bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane are internal standard compounds recommended for volatile priority pollutant determinations (*2*). The compounds benzene-$d_6$ toluene-$d_8$ were added for approximating the concentrations of aromatic nonpriority pollutants. The estimated concentrations included in Tables II and III were calculated by comparing the base peaks of the compounds to the base peak of toluene-$d_8$ and approximating the response factor to be 1.

A 10-g sample of fish tissue (large mouth bass taken from the San Joaquin River, CA) was spiked to 10 ppb with the volatile priority pollutants. The spiked sample was vacuum distilled and the distillate analyzed by GC/MS. The results are shown in Figure 6 and Table IV. All of the volatile priority pollutants, with the exception of acrolein, were detected and identified via a forward search of the EPA/NIH mass spectral library. Coelution of major contaminants (propanal and 3-buten-2-ol) did not interfere with identification of the volatile priority pollutants. The extracted ion mass chromatograms of the coeluting compounds are shown in Figures 7 and 8. The detection and identification of the volatile priority pollutants in the fish distillate indicate that the vacuum distillation procedure can be adapted with FSCC for routine determinations of the volatile priority pollutants in fish tissue.

Average retention time, standard deviation ($\sigma$), and the reverse search retention time window ($2\sigma$) necessary for detection of the internal standard with 95% confidence for each of the internal standards are shown in Table V. The results were obtained by averaging the retention times of the compounds in seven analyses (two standard, two blank, and three fish analysis).

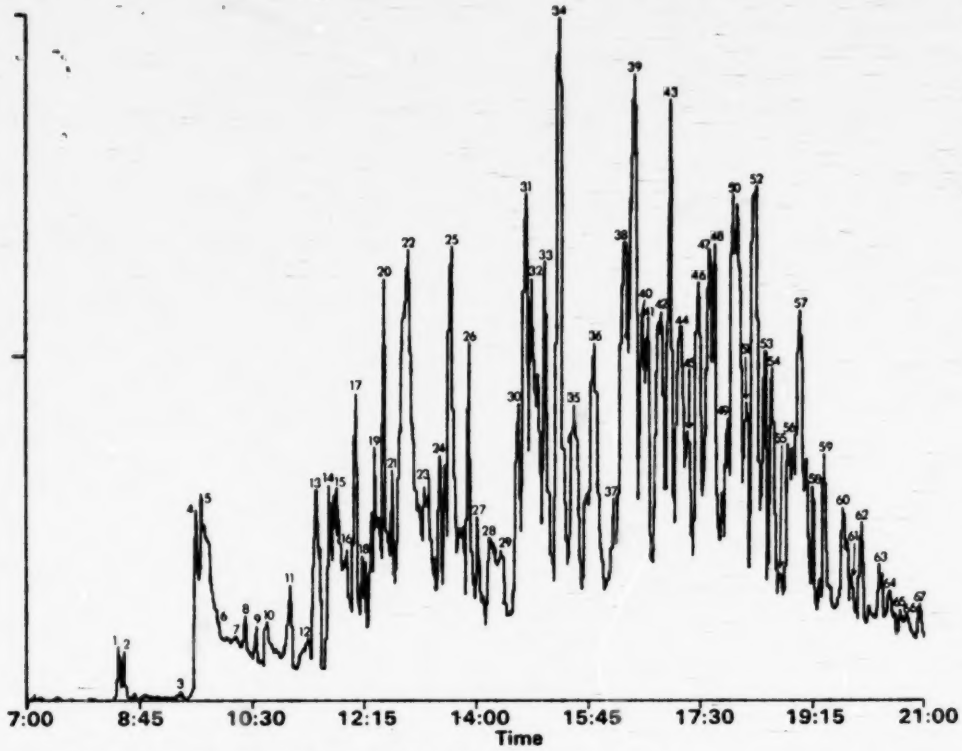


**Figure 5.** Chromatogram of a vacuum distillate prepared from a 10-g carp sample.

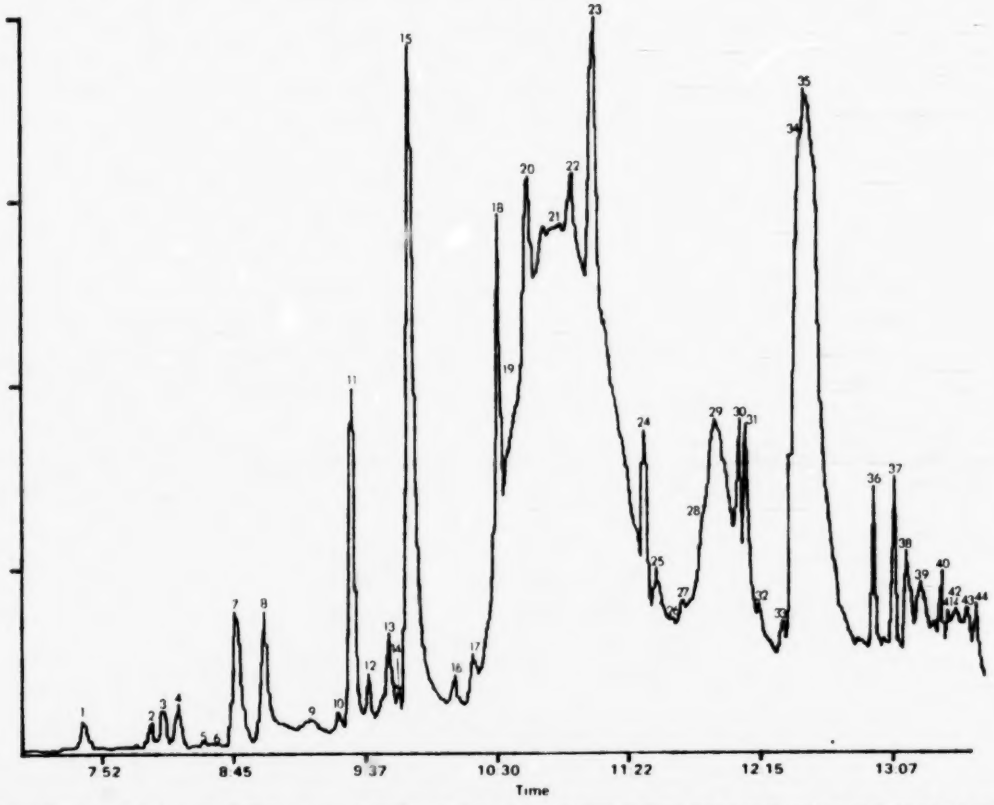


**Figure 6.** Chromatogram of a vacuum distillate prepared from a 10-g large mouth bass sample spiked with 100 ng of each of the volatile priority pollutants.

**Table II. Identification of Peaks in Figure 4**

| no. | formula | retention time[a] (min:s) | mol wt | identification[b] | est concn,[c] ppb |
|---|---|---|---|---|---|
| 1 | $CCl_2F_2$ | 7:08 | 120 | dichlorodifluoromethane | |
| 2 | $CH_3Cl$ | 7:42 | 50 | chloromethane | |
| 3 | $C_4H_8$ | 8:11 | 56 | 2-methyl-1-propene | |
| 4 | $C_4H_6$ | 8:18 | 54 | 1,3-butadiene | |
| 5 | $C_3H_6O$ | 9:31 | 58 | propanal | |
| 6 | $CH_2Cl_2$ | 10:00 | 84 | dichloromethane | |
| 7 | $C_3H_6O$ | 10:04 | 58 | propanal | |
| 8 | $C_6H_{14}$ | 10:27 | 86 | 3-methylpentane | |
| 9 | $C_5H_{10}O$ | 10:37 | 86 | 2-methylbutanal | |
| 10 | $C_5H_6O$ | 10:44 | 82 | 2-methylfuran | |
| 11 | $CH_2ClBr$ | 10:54 | 128 | bromochloromethane | 8 |
| 12 | $C_6H_{12}$ | 11:00 | 84 | 2-methyl-1-pentene | |
| 13 | $C_2HCl_3$ | 11:14 | 132 | 1,1,1-trichloroethene | |
| 14 | $C_6D_6$ | 11:25 | 84 | benzene-$d_6$ | 8 |
| | $C_6H_6$ | 11:27 | 78 | benzene | |
| 15 | $C_7H_{16}$ | 11:31 | 100 | 3-methylhexane | |
| 16 | $C_4H_8O$ | 11:47 | 72 | 3-buten-2-ol | |
| 17 | $C_7H_{14}$ | 12:11 | 98 | methylcyclohexane | 2 |
| 18 | $C_7H_{14}$ | 12:17 | 98 | ethylcyclopentane | |
| 19 | $C_5H_8O$ | 12:31 | 84 | 2,3-dihydro-4-methylfuran | |
| 20 | $C_7D_8$ | 12:38 | 100 | toluene-$d_8$ | 8 |
| | $C_7H_8$ | 12:41 | 92 | toluene | |
| 21 | $C_8H_{12}O$ | 12:50 | 100 | 1,3-dimethylcyclohexane | |
| 22 | $C_6H_{12}O$ | 13:03 | 100 | 2-methylcyclopentanol | 90 |
| | $C_8H_{14}$ | 13:11 | 110 | 3-methyl-1,4-heptadiene | |
| 23 | $C_2Cl_4$ | 13:15 | 164 | tetrachloroethene | |
| | $C_8H_{14}$ | 13:17 | 110 | 3-methyl-1,4-heptadiene | |
| 24 | $C_9H_{18}$ | 13:31 | 126 | 1,2,4-trimethylcyclohexane | |
| 25 | $C_8H_{16}$ | 13:34 | 112 | ethylcyclohexane | 1 |
| 26 | $C_5H_{10}N_2$ | 13:48 | 98 | 4,5-dihydro-2,4-dimethylimidazole | |
| 27 | $C_8H_{10}$ | 14:02 | 106 | ethylbenzene | |
| 28 | $C_8H_{10}$ | 14:12 | 106 | *m* + *p*-xylene | 50 |
| 29 | $C_9H_{18}$ | 14:22 | 126 | 1,2,4-trimethylcyclohexane | |
| 30 | $C_9H_{16}O$ | 14:30 | 140 | 8-nonen-2-one | |
| 31 | $C_9H_{20}$ | 14:38 | 128 | nonane | |
| 32 | $C_8H_{10}$ | 14:43 | 106 | *o*-xylene | |
| 33 | $C_4H_8Cl_2$ | 14:58 | 126 | 1,4-dichlorobutane | |
| 34 | $C_9H_{12}$ | 15:20 | 120 | (1-methylethyl)benzene | |
| 35 | $C_9H_{18}$ | 15:28 | 126 | propylcyclohexane | 1 |
| 36 | $C_{10}H_{16}$ | 15:34 | 136 | 4-carene | |
| 37 | $C_9H_{12}$ | 15:55 | 120 | propylbenzene | 2 |
| 38 | $C_9H_{12}$ | 16:05 | 120 | 1-ethyl-3-methylbenzene | 40 |
| 39 | $C_9H_{12}$ | 16:12 | 120 | 1,3,5-trimethylbenzene | |
| 40 | $C_9H_{12}$ | 16:28 | 120 | 1-ethyl-4-methylbenzene | |
| 41 | $C_9H_{12}$ | 16:44 | 120 | 1,3,5-trimethylbenzene | 50 |
| 42 | $C_{10}H_{14}$ | 17:00 | 134 | (2-methylpropyl)benzene | |
| 43 | $C_{10}H_{14}$ | 17:04 | 134 | (1-methylpropyl)benzene | |
| 44 | $C_{10}H_{14}$ | 17:12 | 134 | 1-methyl-3-propylbenzene | |
| 45 | $C_9H_{12}$ | 17:19 | 120 | 1,2,3-trimethylbenzene | |
| 46 | $C_9H_{10}$ | 17:38 | 118 | indan | |
| 47 | $C_{10}H_{14}$ | 17:47 | 134 | 1-methyl-4-propylbenzene | 10 |
| 48 | $C_{10}H_{14}$ | 17:53 | 134 | 2-ethyl-1,4-dimethylbenzene | 10 |
| 49 | $C_7H_{10}O$ | 18:01 | 110 | 2,2-dimethyl-3,4-pentadienal | |
| 50 | $C_{10}H_{14}$ | 18:07 | 134 | 1-methyl-4-propylbenzene | |
| 51 | $C_{10}H_{14}$ | 18:16 | 134 | 2-ethyl-1,2-dimethylbenzene | |
| 52 | $C_{10}H_{14}$ | 18:26 | 134 | 4-ethyl-1,2-dimethylbenzene | 9 |
| 53 | $C_{10}H_{12}$ | 18:33 | 132 | 1-methylindan | |
| 54 | $C_{10}H_{14}$ | 18:51 | 134 | 1-ethyl-3,5-dimethylbenzene | |
| 55 | $C_{10}H_{14}$ | 19:00 | 134 | 1,2,3,5-tetramethylbenzene | |
| 56 | $C_{10}H_{14}$ | 19:06 | 134 | 1,2,4,5-tetramethylbenzene | |
| 57 | $C_{11}H_{16}$ | 19:23 | 148 | (1-ethylpropyl)benzene | |
| 58 | $C_{10}H_{12}$ | 19:30 | 132 | 4-methylindan | 2 |
| 59 | $C_{10}H_{12}$ | 19:42 | 132 | 1-methylindan | |
| 60 | $C_{11}H_{16}$ | 19:51 | 148 | *p*-isobutyltoluene | |
| 61 | $C_{10}H_{12}$ | 19:58 | 132 | 1,2,3,4-tetrahydronaphthalene | |
| 62 | $C_{11}H_{16}$ | 20:12 | 148 | 1-ethyl-3-(1-methylethyl)benzene | |
| 63 | $C_{11}H_{14}$ | 20:17 | 146 | 1,1-dimethylindan | |
| 64 | $C_{10}H_8$ | 20:23 | 128 | naphthalene | 1 |
| | $C_{10}H_{12}O$ | 20:23 | 148 | 1-(4-ethylphenyl)ethanone | |
| 65 | $C_{11}H_{16}$ | 20:35 | 148 | ethyl-1,2,4-trimethylbenzene | |
| 66 | $C_{12}H_{18}$ | 20:45 | 162 | *p*-(1-ethylpropyl)toluene | |

[a] 30 m DB5 fused silica capillary column (0.32 mm bore, 1.0 μm film thickness) preceded by 18 in. of $^1/_8$ in. o.d. stainless steel tubing. Carrier gas helium at 2 mL/min. Temperature program: 3 min isothermal at −99 °C, then 30 °C/min to 70 °C, then 20 °C/min to 180 °C. Hold at 180 °C for 10 min. [b] Identifications were assisted by computerized search of the EPA/NIH mass spectral library. [c] The estimated concentrations were calculated by comparing the base peaks of the compounds to the base peak of toluene-$d_8$ and approximating the response factor to be 1.

Table III. Identification of Peaks in Figure 5

| no. | formula | retention time[a] (min:s) | mol wt | identification[b] | est concn,[c] ppb |
|---|---|---|---|---|---|
| 1 | $C_4H_8$ | 8:26 | 56 | 2-butene | |
| 2 | $C_4H_6$ | 8:31 | 54 | 1,3-butadiene | |
| 3 | $C_4H_8O$ | 9:23 | 72 | 2-methyl-2-propen-1-ol | |
| 4 | $C_4H_8O$ | 9:39 | 72 | 2-buten-1-ol | |
| 5 | $C_5H_8$ | 9:44 | 68 | 3-methyl-1,2-butadiene | |
| 6 | $C_5H_6$ | 10:07 | 66 | 1,3-cyclopentadiene | |
| 7 | $CH_2Cl_2$ | 10:15 | 84 | dichloromethane | |
| 8 | $C_5H_{10}O$ | 10:24 | 86 | 1-methoxy-1-butene | |
| 9 | $C_6H_{14}$ | 10:34 | 86 | 3-methylpentane | |
| 10 | $C_6H_{14}$ | 10:45 | 86 | 3-methylpentane | |
| 11 | $C_6H_{14}O$ | 11:07 | 102 | 3-methyl-1-pentanol | |
| 12 | $C_6H_{10}$ | 11:22 | 82 | (1-methylethenyl)cyclopropane | |
| 13 | $C_6D_6$ | 11:30 | 84 | benzene-$d_6$ | 8 |
| | $C_6H_6$ | 11:31 | 78 | benzene | |
| 14 | $C_7H_{14}$ | 11:45 | 98 | 1,3-dimethylcyclopentane | |
| 15 | $C_7H_{14}O$ | 11:51 | 114 | 4-methylcyclohexanol | |
| 16 | $C_7H_{12}$ | 11:59 | 96 | 1,4-dimethyl-1,2-pentadiene | |
| 17 | $C_7H_{14}$ | 12:08 | 98 | methylcyclohexane | |
| 18 | $C_7H_{14}$ | 12:13 | 98 | ethylcyclopentane | |
| 19 | $C_8H_{18}$ | 12:27 | 114 | 3,3-dimethylhexane | |
| 20 | $C_7D_8$ | 12:35 | 100 | toluene-$d_8$ | 8 |
| | $C_7H_8$ | 12:36 | 92 | toluene | |
| 21 | $C_8H_{16}$ | 12:41 | 112 | 1,2-dimethylcyclohexane | |
| 22 | $C_8H_{12}O$ | 12:57 | 100 | 2-methylcyclopentanol | |
| 23 | $C_9H_{18}$ | 13:13 | 126 | 1,2,4-trimethylcyclohexane | |
| 24 | $C_9H_{18}$ | 13:26 | 126 | 1,1,2-trimethylcyclohexane | |
| 25 | $C_8H_{10}$ | 13:36 | 106 | *m,p*-xylene | 120 |
| 26 | $C_8H_{10}$ | 13:54 | 106 | *o*-xylene | |
| 27 | $C_9H_{16}$ | 14:01 | 126 | 1-ethyl-1-methylcyclohexane | |
| 28 | $C_9H_{10}$ | 14:13 | 124 | indan | |
| 29 | $C_{10}H_{16}$ | 14:24 | 136 | 4-carene | |
| 30 | $C_9H_{12}$ | 14:40 | 120 | propylbenzene | |
| 31 | $C_9H_{12}$ | 14:47 | 120 | 1-ethyl-3-methylbenzene | 130 |
| 32 | $C_9H_{12}$ | 14:54 | 120 | 1,3,5-trimethylbenzene | |
| 33 | $C_9H_{12}$ | 15:05 | 120 | 1,2,4-trimethylbenzene | |
| 34 | $C_9H_{12}$ | 15:20 | 120 | 1,2,3-trimethylbenzene | 300 |
| 35 | $C_9H_{10}$ | 15:34 | 134 | methylbenzene acetaldehyde | |
| 36 | $C_{10}H_{16}$ | 15:54 | 136 | 1-methyl-4-(1-methylethenyl)cyclohexene | |
| 37 | $C_9H_{10}$ | 16:09 | 118 | 1-ethenyl-2-methylbenzene | |
| 38 | $C_{10}H_{14}$ | 16:21 | 134 | 1,2-diethylbenzene | |
| 39 | $C_{10}H_{14}$ | 16:31 | 134 | 1-ethyl-2,3-dimethylbenzene | 120 |
| 40 | $C_{10}H_{18}$ | 16:37 | 138 | decalin | |
| 41 | $C_{10}H_{14}$ | 16:42 | 134 | (1-methylpropyl)benzene | |
| 42 | $C_{10}H_{14}$ | 16:53 | 134 | 1,4-diethylbenzene | |
| 43 | $C_9H_{10}O$ | 17:04 | 134 | 2,5-dimethylbenzaldehyde | |
| 44 | $C_{10}H_{12}$ | 17:10 | 132 | 2-methylindan | |
| 45 | $C_{11}H_{16}$ | 17:18 | 148 | *p*-isobutyltoluene | |
| 46 | $C_{10}H_{14}$ | 17:28 | 134 | 1,3-diethylbenzene | 40 |
| 47 | $C_{11}H_{20}$ | 17:42 | 152 | 2-methyldecalin | |
| 48 | $C_9H_{10}O$ | 17:45 | 134 | 3,4-dimethylbenzaldehyde | |
| 49 | $C_{11}H_{14}$ | 17:53 | 146 | 1,6-dimethylindan | |
| 50 | $C_{11}H_{20}$ | 18:00 | 152 | 2-methyldecalin | |
| 51 | $C_{11}H_{16}$ | 18:14 | 148 | *p*-isobutyltoluene | |
| 52 | $C_{10}H_{12}$ | 18:22 | 132 | 5-methylindan | 60 |
| 53 | $C_{11}H_{16}$ | 18:33 | 148 | diethylmethylbenzene | |
| 54 | $C_{11}H_{16}$ | 18:39 | 148 | (1,1-dimethylpropyl)benzene | |
| 55 | $C_{12}H_{18}$ | 18:53 | 162 | *p*-(1-ethylpropyl)toluene | |
| 56 | $C_{11}H_{14}$ | 18:57 | 146 | 1,1-dimethylindan | |
| 57 | $C_{11}H_{16}$ | 19:04 | 148 | 1-ethyl-3-(1-methylethyl)benzene | 80 |
| 58 | $C_{11}H_{16}$ | 19:16 | 148 | 1-ethyl-2,4,5-trimethylbenzene | |
| 59 | $C_{12}H_{18}$ | 19:28 | 162 | 2,4-dimethyl-1-(1-methylpropyl)benzene | |
| 60 | $C_{12}H_{22}$ | 19:42 | 166 | 2,6-dimethyldecalin | |
| 61 | $C_{12}H_{16}$ | 19:52 | 160 | 1,4,7-trimethylindan | 10 |
| 62 | $C_{11}H_{14}$ | 20:02 | 146 | (3-methyl-2-butenyl)benzene | 30 |
| 63 | $C_{11}H_{14}$ | 20:18 | 146 | 5,6-dimethylindan | |
| 64 | $C_{12}H_{18}$ | 20:28 | 162 | 1,3,5-triethylbenzene | 20 |
| 65 | $C_{11}H_{14}$ | 20:39 | 146 | 4,6-dimethylindan | |
| 66 | $C_{12}H_{16}$ | 20:45 | 160 | 1,4,7-trimethylindan | |
| 67 | $C_{11}H_{10}$ | 20:57 | 142 | 2-methylnaphthalene | 10 |

[a] 30 m DB5 fused silica capillary column (0.32 mm bore, 1.0 μm film thickness) preceded by 18 in. of 1/8 in. o.d. stainless steel tubing. Carrier gas helium at 2 mL/min. Temperature program: 3 min isothermal at −99 °C, then 30 °C/min to 70 °C, then 20 °C/min to 180 °C. Hold at 180 °C for 10 min. [b] Identifications were assisted by a computerized search of the EPA/NIH mass spectral library. [c] The estimated concentrations were calculated by comparing the base peaks of the compounds to the base peak of toluene-$d_8$ and approximating the response factor to be 1.

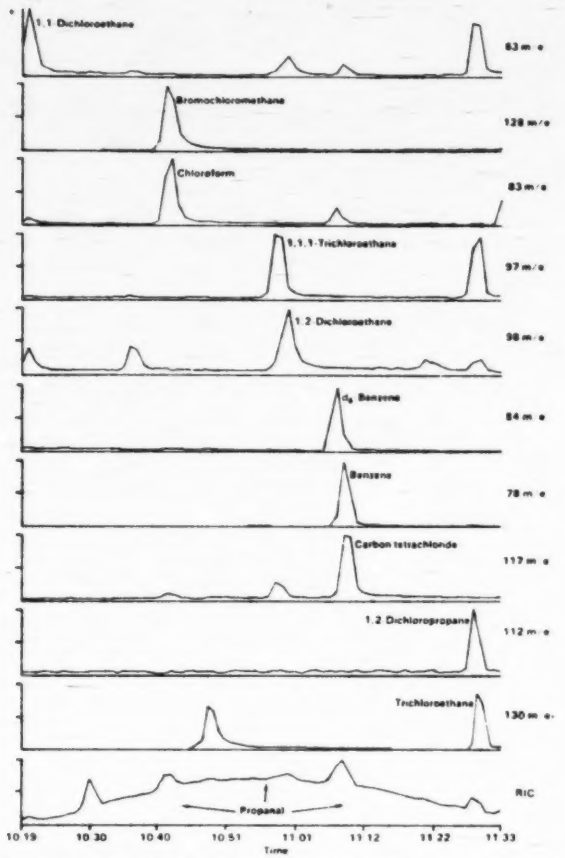


**Figure 7.** Mass chromatograms of volatile priority pollutants that coelute with propanal in Figure 5.

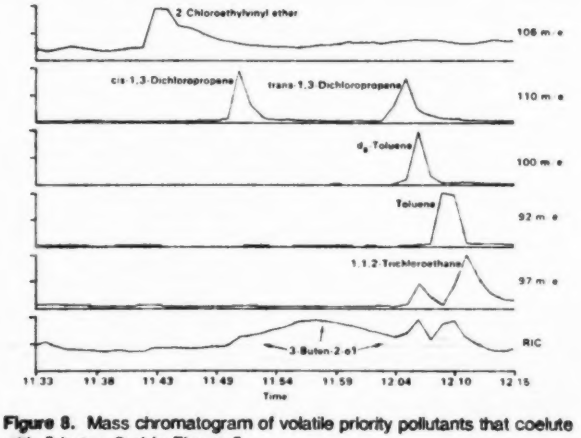


**Figure 8.** Mass chromatogram of volatile priority pollutants that coelute with 3-buten-2-ol in Figure 5.

The vacuum distillation technique requires approximately 35 min and samples can be analyzed in succession at 45-min intervals. This includes ample time (10 min) for preparation of the vacuum distillation apparatus for the next sample.

The sample storage vessels, which also serve as distillation sample chambers, simplify sample manipulation. Samples are easily placed in vessels that are sealed with O-ring fittings for storage. A sample can be removed from storage and immediately connected to the apparatus for distillation. With minimum sample aliquot handling, the potential for contamination of the sample by the laboratory environment is reduced.

**Table IV. Identification of Peaks in Figure 6**

| no. | formula | retention time[a] (min:s) | mol wt | identification[b] |
|---|---|---|---|---|
| 1 | $CH_3Cl$ | 7:45 | 50 | chloromethane |
| 2 | $C_2H_3Cl$ | 8:12 | 62 | vinyl chloride |
| 3 | $C_4H_8$ | 8:18 | 56 | 2-butene |
| 4 | $C_4H_6$ | 8:23 | 54 | 1,3-butadiene |
| 5 | $C_4H_8$ | 8:32 | 56 | 2-butene |
| 6 | $C_4H_4$ | 8:38 | 52 | 1-buten-3-yne |
| 7 | $CH_3Br$ | 8:46 | 94 | bromomethane |
| 8 | $C_2H_5Cl$ | 8:58 | 64 | chloroethane |
| 9 | $C_3H_9N$ | 9:15 | 59 | *N,N*-dimethylmethanamine |
| 10 | $CCl_3F$ | 9:24 | 136 | trichlorofluoromethane |
| | $C_5H_{10}$ | 9:27 | 70 | 1,1-dimethylcyclopropane |
| 11 | $C_3H_6O$ | 9:32 | 72 | 2-buten-1-ol |
| 12 | $C_5H_{10}$ | 9:39 | 70 | 2-methyl-2-butene |
| 13 | $C_2H_2Cl_2$ | 9:46 | 96 | 1,1-dichloroethene |
| | $CH_3I$ | 9:47 | 142 | iodomethane |
| 14 | $C_3H_3N$ | 9:50 | 53 | acrylonitrile |
| | $C_5H_8$ | 9:50 | 68 | cyclopentene |
| 15 | $CH_2Cl_2$ | 9:55 | 84 | dichloromethane |
| | $C_5H_6$ | 9:58 | 66 | 1,3-cyclopentadiene |
| 16 | $C_2H_2Cl_2$ | 10:13 | 96 | *trans*-1,2-dichloroethene |
| 17 | $C_2H_4Cl_2$ | 10:21 | 98 | 1,1-dichloroethane |
| 18 | $C_5H_{10}O$ | 10:30 | 86 | 2-methylbutanal |
| 19 | $C_5H_6O$ | 10:34 | 82 | 2-methylfuran |
| 20 | $CH_2ClBr$ | 10:42 | 128 | bromochloromethane |
| | $CHCl_3$ | 10:42 | 118 | trichloromethane |
| 21 | $C_3H_6O$ | 10:35 | 58 | propanal |
| 22 | $C_2H_3Cl_3$ | 10:58 | 132 | 1,1,1-trichloroethane |
| | $C_2H_4Cl_2$ | 11:00 | 98 | 1,2-dichloroethane |
| 23 | $C_6D_6$ | 11:08 | 84 | benzene-$d_6$ |
| | $C_6H_6$ | 11:09 | 78 | benzene |
| | $CCl_4$ | 11:09 | 152 | carbon tetrachloride |
| 24 | $C_2HCl_3$ | 11:29 | 130 | trichloroethene |
| | | 11:30 | 112 | 1,2-dichloropropane |
| 25 | $CHCl_2Br$ | 11:34 | 162 | bromodichloromethane |
| 26 | $C_3H_7I$ | 11:40 | 170 | 2-iodopropane |
| 27 | $C_4H_7OCl$ | 11:45 | 106 | 2-chloroethyl vinyl ether |
| 28 | $C_3H_4Cl_2$ | 11:51 | 110 | *cis*-1,3-dichloropropene |
| 29 | $C_4H_8O$ | 11:57 | 72 | 3-buten-2-ol |
| 30 | $C_7D_8$ | 12:07 | 100 | toluene-$d_8$ |
| | $C_3H_4Cl_2$ | 12:07 | 110 | *trans*-1,3-dichloropropene |
| 31 | $C_7H_8$ | 12:09 | 92 | toluene |
| | $C_2H_3Cl_3$ | 12:11 | 132 | 1,1,2-trichloroethane |
| 32 | $C_3H_6ClBr$ | 12:15 | 156 | 2-bromo-1-chloropropane |
| 33 | $CHClBr_2$ | 12:28 | 206 | dibromochloromethane |
| 34 | $C_6H_{12}O$ | 12:33 | 100 | 2-methylcyclopentanol |
| 35 | $C_2Cl_4$ | 12:37 | 164 | tetrachloroethene |
| 36 | $C_6H_5Cl$ | 13:00 | 112 | chlorobenzene |
| 37 | $C_8H_{10}$ | 13:09 | 106 | ethylbenzene |
| 38 | $C_8H_{10}$ | 13:13 | 106 | *m* + *p*-xylene |
| 39 | $C_8H_{16}O$ | 13:19 | 128 | 2-methyl-1,4-heptanone |
| 40 | $CHBr_3$ | 13:26 | 250 | bromoform |
| 41 | $C_8H_{10}$ | 13:30 | 106 | *o*-xylene |
| 42 | $C_6H_{12}O$ | 13:33 | 114 | 4-methylcyclohexanol |
| 43 | $C_4H_8Cl_2$ | 13:37 | 126 | 1,4-dichlorobutane |
| 44 | $C_2H_2Cl_4$ | 13:41 | 166 | 1,1,2,2-tetrachloroethane |

[a] 30 m DB5 fused silica capillary column (0.32 mm bore, 1.0 μm film thickness) preceded by 18 in. of 1/8 in. o.d. stainless steel tubing. Carrier gas helium at 2 mL/min. Temperature program: 3 min isothermal at −99 °C, then 30 °C/min to 70 °C, then 20 °C/min to 180 °C. Hold at 180 °C for 10 min. [b] Identifications were assisted by computerized search of the EPA/NIH mass spectral library.

The large number of organic compounds than can be analyzed with FSCC by this technique is illustrated in Figures 4 and 5 and in Tables II and III. The lowest pressure that can be obtained in the distillation apparatus is limited to the vapor pressure of water at −20 °C (the temperature of the water condensation trap C1) which is 0.78 torr (5). The

**Table V. Fused Silica Capillary Column Retention Times for Internal Standards Concentrated by Vacuum Distillation**

| formula | retention time[a] (min:s) | 1σ std dev | window width (2σ std dev)[b] |
|---|---|---|---|
| bromochloromethane | 10:52 | 20 s | 40 s |
| benzene-$d_6$ | 11:14 | 26 s | 52 s |
| toluene-$d_8$ | 12:29 | 28 s | 56 s |
| 2-bromo-1-chloropropane | 12:38 | 30 s | 60 s |
| 1,4-dichlorobutane | 14:10 | 66 s | 132 s |

[a] 30 m DB5 fused silica capillary column (0.32 mm bore, 1.0 μm film thickness) preceded by 18 in. of 1/8 in. o.d. stainless steel tubing. Carrier gas helium at 2 mL/min. Temperature program: 3 min isothermal at −99 °C, then 30 °C/min to 70 °C, then 20 °C/min to 180 °C. Hold at 180 °C for 10 min. [b] Standard deviation were computed by using retention times of the compounds for eight analyses (three standard solution distillates, two background distillates, and three fish distillates).

**Table VI. Vapor Pressures[a] of Identified Vacuum Distilled Compounds**

| compounds | vapor pressure in torr −20 °C | +20 °C | +50 °C |
|---|---|---|---|
| acrylonitrile | 9.830 | 84.89 | 301.2 |
| benzene | 6.28 | 57.36 | 201.3 |
| chlorobenzene | 0.601 | 9.03 | 38.35 |
| chloromethane | 801.5 | 3449 | 8128 |
| 1,4-diethylbenzene | NC[b] | 1.106 | 6.14 |
| ethylbenzene | 1.21[c] | 7.47 | 32.92 |
| glycol | NC | NC | 0.976[d] |
| isopropylbenzene | NC | 3.345 | 17.4 |
| naphthalene | NC | 0.053 | 0.805 |
| *n*-heptane | 3.34 | 32.75 | 125.2 |
| tetrachloroethene | 1.11 | 13.65 | 59.60 |
| 1,1,2,2-tetrachloroethane | NC | 4.75 | 23.12 |
| toluene | 2.037 | 20.92 | 82.21 |
| *trans*-1,2-dimethylcyclohexane | 1.19 | 14.05 | 59.95 |
| 1,3,5-triethylbenzene | NC | NC | 3.38 |
| | NC | NC | 2.00 |
| ethylbenzene | | | |
| water | 0.776 | 17.535 | 92.51 |

[a] Vapor pressures are derived from tables in CRC "Handbook of Chemistry and Physics", 1972. [b] Could not be calculated from tables. [c] Calculated for −10 °C. [d] Calculated for 53 °C.

physical limitation for compounds that can be detected is that the vapor pressure of the compound must be greater than 0.78 torr in the sample chamber (~50 °C). This concept is supported by comparing the compounds identified in actual samples with their respective vapor pressures at 50 °C (see Table VI).

It is also of interest to note that naphthalene (~1 ppb), a semivolatile priority pollutant, is detected (Table II). The detection of naphthalene in the vacuum distillate is important since naphthalene is presently analyzed as a semivolatile priority pollutant after liquid extraction and concentration (6). Liquid extraction yields a final extract (1 mL) that represents an initial 20-g fish sample. Only 2 μL of the extract, which represents 40 mg of sample, can be injected onto a capilary column. Comparing a 40-mg sample-equivalent injection to the 10-g sample-equivalent distillate obtained by vacuum distillation, it is apparent that the distillate can obtain sensitivities 250 times greater for many semivolatile priority pollutant determinations. The 1 ppb naphthalene detected is 500 times lower than the GC/MS detection limit reported in the U.S. EPA extraction procedure (6).

This technique has potential to greatly increase GC/MS analytical sensitivities and reduce the time required for analysis of volatile compounds in fish tissue. The attributes of this technique justify further investigation to validate the technique for quantitative determinations of the volatile compounds in other media.

**Registry No.** Chloromethane, 74-87-3; vinyl chloride, 75-01-4; bromomethane, 74-83-9; chloroethane, 75-00-3; trichlorofluoromethane, 75-69-4; acrolein, 107-02-8; acetone, 67-64-1; 1,1-dichloroethene, 75-35-4; acrylonitrile, 107-13-1; dichloromethane, 75-09-2; *trans*-1,2-dichloroethene, 156-60-5; 1,1-dichloroethane, 75-34-3; chloroform, 67-66-3; 1,1,1-trichloroethane, 71-55-6; 1,2-dichloroethane, 107-06-2; benzene, 71-43-2; carbon tetrachloride, 56-23-5; trichloroethene, 79-01-6; 1,2-dichloropropane, 78-87-5; bromodichloromethane, 75-27-4; 2-chloroethyl vinyl ether, 110-75-8; *cis*-1,3-dichloropropene, 10061-01-5; *trans*-1,3-dichloropropene, 10061-02-6; toluene, 108-88-3; 1,1,2-trichloroethane, 79-00-5; dibromochloromethane, 124-48-1; tetrachloroethene, 127-18-4; chlorobenzene, 108-90-7; ethylbenzene, 100-41-4; *m*-xylene, 108-38-3; bromoform, 75-25-2; *o*-xylene, 95-47-6; 1,1,2,2-tetrachloroethane, 79-34-5; *p*-xylene, 106-42-3; dichlorodifluoromethane, 75-71-8; 2-methyl-1-propene, 115-11-7; 1,3-butadiene, 106-99-0; propanal, 123-38-6; 3-methylpentane, 96-14-0; 2-methylbutanal, 96-17-3; 2-methylfuran, 534-22-5; 2-methyl-1-pentene, 763-29-1; 3-methylhexane, 589-34-4; 3-buten-2-ol, 598-32-3; methylcyclohexane, 108-87-2; ethylcyclopentane, 1640-89-7; 2,3-dihydro-4-methylfuran, 34314-83-5; 1,3-dimethylcyclohexane, 591-21-9; 2-methylcyclopentanol, 24070-77-7; 3-methyl-1,4-heptadiene, 1603-01-6; 1,2,4-trimethylcyclohexane, 2234-75-5; ethylcyclohexane, 1678-91-7; 4,5-dihydro-2,4-dimethylimidazole, 930-61-0; 8-nonen-2-one, 5009-32-5; nonane, 111-84-2; (1-methylethyl)benzene, 98-82-8; propylcyclohexane, 1678-92-8; 4-carene, 29050-33-7; propylbenzene, 103-65-1; 1-ethyl-3-methylbenzene, 620-14-4; 1,3,5-trimethylbenzene, 108-67-8; 1-ethyl-4-methylbenzene, 622-96-8; (2-methylpropyl)benzene, 538-93-2; (1-methylpropyl)benzene, 135-98-8; 1-methyl-3-propylbenzene, 1074-43-7; 1,2,3-trimethylbenzene, 526-73-8; indan, 496-11-7; 1-methyl-4-propylbenzene, 1074-55-1; 2-ethyl-1,4-dimethylbenzene, 1758-88-9; 2,2-dimethyl-3,4-pentadienal, 4058-51-9; 4-ethyl-1,2-dimethylbenzene, 934-80-5; 1-methylindan, 767-58-8; 1-ethyl-3,5-dimethylbenzene, 934-74-7; 1,2,3,5-tetramethylbenzene, 527-53-7; 1,2,4,5-tetramethylbenzene, 95-93-2; (1-ethylpropyl)-benzene, 1196-58-3; 4-methylindan, 824-22-6; *p*-isobutyltoluene, 5161-04-6; 1,2,3,4-tetrahydronaphthalene, 119-64-2; 1-ethyl-3-(1-methylethyl)benzene, 4920-99-4; 1,1-dimethylindan, 4912-92-9; naphthalene, 91-20-3; 1-(4-ethylphenyl)ethanone, 937-30-4; ethyl-1,2,4-trimethylbenzene, 54120-62-6; *p*-(1-ethylpropyl)toluene, 22975-58-2; 2-butene, 107-01-7; 2-methyl-2-propen-1-ol, 513-42-8; 2-buten-1-ol, 6117-91-5; 3-methyl-1,2-butadiene, 598-25-4; 1,3-cyclopentadiene, 542-92-7; 1-methoxy-1-butene, 29512-02-5; 3-methyl-1-pentanol, 589-35-5; (1-methylethenyl)cyclopropane, 4663-22-3; 1,3-dimethylcyclopentane, 2453-00-1; 4-methylcyclohexanol, 589-91-3; 1,4-dimethyl-1,2-pentadiene, 69153-20-4; 3,3-dimethylhexane, 563-16-6; 1,2-dimethylcyclohexane, 583-57-3; 1,1,2-trimethylcyclohexane, 7094-26-0; 1-ethyl-1-methylcyclohexane, 4926-90-3; 1,2,4-trimethylbenzene, 95-63-6; 1,2,3-trimethylbenzene, 526-73-8; methylbenzene acetaldehyde, 69380-02-5; 1-methyl-4-(1-methylethenyl)cyclohexene, 138-86-3; 1-ethenyl-2-methylbenzene, 611-15-4; 1,2-diethylbenzene, 135-01-3; 1-ethyl-2,3-dimethylbenzene, 933-98-2; 1,4-diethylbenzene, 105-05-5; 2,5-dimethylbenzaldehyde, 5779-94-2; 2-methylindan, 824-63-5; 1,3-diethylbenzene, 141-93-5; 2-methyldecalin, 2958-76-1; 3,4-dimethylbenzaldehyde, 5973-71-7; 1,6-dimethylindan, 17059-48-2; 5-methylindan, 874-35-1; diethylmethylbenzene, 25550-13-4; (1,1-dimethylpropyl)benzene, 2049-95-8; 1-ethyl-2,4,5-trimethylbenzene, 17851-27-3; 2,4-dimethyl-1-(1-methylpropyl)benzene, 1483-60-9; 2,6-dimethyldecalin, 1618-22-0; 1,4,7-trimethylindan, 54340-87-3; (3-methyl-2-butenyl)benzene, 4489-84-3; 5,6-dimethylindan, 1075-22-5; 1,3,5-triethylbenzene, 102-25-0; 4,6-dimethylindan, 1685-82-1; 2-methylnaphthalene, 91-57-6; 1-buten-3-yne, 689-97-4; *N,N*-dimethylmethanamine, 75-50-3; 1,1-dimethylcyclopropane, 1630-94-0; 2-methyl-2-butene, 513-35-9; iodomethane, 74-88-4; cyclopentene, 142-29-0; tri-

chloromethane, 67-66-3; 2-iodopropane, 75-30-9; 2-methyl-4-heptanone, 626-33-5; 3-ethyl-1,2-dimethylbenzene, 933-98-2.

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